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FURTHER STUDIES OF DELETERIOUS SUBSTANCES IN INDIANA AGGREGATES

JULY 1959
NO. 18

by

R. L. SCHUSTER

Joint Highway Research Project

PURDUE UNIVERSITY
LAFAYETTE INDIANA

PROGRESS REPORT NO. 2

FURTHER STUDIES OF DELETERIOUS SUBSTANCES
IN INDIANA AGGREGATES

TO: K. B. Woods, Director
Joint Highway Research Project

July 1, 1959

FROM: H. L. Michael, Assistant Director

File: 5-9-6
Project C-36-42F

Attached is a report entitled, "Progress Report No. 2 - Further Studies of Deleterious Substances in Indiana Aggregates". It has been prepared by Mr. R. L. Schuster, Research Assistant on our staff under the supervision of Dr. J. F. McLaughlin.

The material reported is part of a research project concerned with the physical properties and freeze-thaw durabilities of deleterious substances. The attached report contains information on shales only. The study of cherts is continuing and will be reported at a later date.

The report is submitted for the record.

Respectifully submitted,

Harold L. Michael

Harold L. Michael, Assistant Director
Joint Highway Research Project

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Progress Report No. 2

FURTHER STUDIES OF DELETERIOUS SUBSTANCES IN INDIANA AGGREGATES

By

R. L. Schuster
Research Assistant

Joint Highway Research Project
File No. 5-9-6
Project No. C-36-42F

Purdue University
Lafayette, Indiana

July 1, 1959

PROGRESS REPORT ON FURTHER STUDIES
OF DELETERIOUS SUBSTANCES IN INDIANA AGGREGATES

INTRODUCTION

On October 23, 1957, a plan of study was approved by the Advisory Board in which a comprehensive study of the physical properties and freeze-thaw durabilities of certain deleterious constituents of Indiana's aggregates was proposed. The purposes of this study were: (a) to learn more about those properties of the deleterious constituents of Indiana's aggregates which relate to the freeze-thaw durability of the concrete in which these deleterious materials are used as part of the aggregate, and to note differences in the properties of deleterious materials which are of the same type but are obtained from different areas; (b) to determine whether Indiana's present specifications on deleterious substances categorize aggregates on a realistic basis; and (c) to suggest, if possible, a simplified version of the present specifications on the basis of the results of the combined tests. The investigation was to be conducted primarily as a study of the durability of concrete produced from different deleterious materials of coarse aggregate size in combination with a standard fine aggregate, a standard crushed stone coarse aggregate (both with good field service records), and a standard portland cement.

It was decided to limit the study to an investigation of shales and cherts because these two rock types are probably the most abundant of the deleterious constituents of Indiana's aggregates, and because there is still much to be learned about the effects of these two materials on the freeze-thaw durability of concrete. The study began

with obtaining aggregate samples of shale and chert from various sources in sufficient quantities to be used in the testing. After sufficient material had been accumulated, the investigation was divided into two closely connected phases; in general the shales being studied first, the cherts second. At present, significant results are available only for the shale portion of the study. Therefore this progress report will be limited to discussion of the tests conducted on the shales and to the results of these tests.

REVIEW OF RESEARCH ON SHALE IN CONCRETE

It has long been suspected that shales have a part in the deterioration of concrete in which they are used as aggregate. In 1927, Lang (1)* showed a direct relationship between per cent of shale in gravel used as concrete aggregate and loss of compressive strength for the resulting concrete. This loss in strength is generally considered to be due to the inherent weakness of the shale particles themselves. The effect of shale on the freeze-thaw durability of concrete is not so clearly defined as its effect on the strength of concrete, however. For quantities of shale up to five per cent, Lang found about two per cent reduction of strength for each one per cent of shale when the concrete was subjected to eleven cycles of freezing and thawing. As a result of his studies, Lang concluded that for pavement concrete the only harmful effect of shale consists of surface pitting of the concrete. In a later report, Lang (2) emphasized the difficulty of evaluating the deleterious effects of shale. He pointed out that the properties of shales vary considerably from one area to another, and that

*Numbers in parentheses refer to the list of references which appears at the end of this report.

the freeze-thaw durability of one shale may differ considerably from that of another shale. He also noted that shales grade into other rock types such as slates or siltstones to further confuse the situation.

The Kentucky State Highway Department also carried out some early studies of the effects of shales on concrete durability (3). They studied the effects of sodium sulfate solutions and of natural weathering on concrete made with varying percentages of a "low grade blue shale" coarse aggregate and concluded the blue shale should be limited in use in concrete to less than five per cent of the coarse aggregate.

The Tennessee State Highway Department carried out a study of the effect of shale in coarse aggregate on the strength of concrete and on its resistance to freezing and thawing. Walker and Proudley (4) note that in this investigation the concrete specimens were made in two groups, one of which was subjected to normal curing conditions and the other to freezing and thawing conditions. The report of this study states that the inclusion of 5 and 10 per cent of shale in the coarse aggregate had no effect on the durability of concrete subjected to 45 cycles of freezing and thawing and no important influence on either the compressive or transverse strength. It was concluded that five per cent would make a suitable specification limit on shale, provided that the total amount of shale and similar substances does not exceed this amount.

Apparently very little has been published on the effects of shale on concrete durability since the early 1930's. Thus present-day specifications for shale in concrete aggregate are based on the fragmentary information provided by the aforementioned studies plus actual service experience in the various states. The consensus of the results of the studies mentioned above seems to be that shale in small percentages

(perhaps as much as 10 per cent) doesn't cause severe damage to concrete exposed to freezing and thawing, but that surface pitting does result from the shale. These studies showed that some shales act differently than others but did not suggest any methods for differentiating "good" shales from "bad" shales.

As early as the 1930's many state highway departments had specifications limiting the percentage of shale allowable in concrete aggregate. Walker and Proudley (4) noted 22 states which limited either shale or "soft, friable fragments and other deleterious particles including shale". Twelve of the states had definite specifications on shale itself in coarse aggregate with most of these allowing from 0.5 to 1.0 per cent shale. Nineteen of the states had specifications on soft, friable fragments including shale with most of these allowing about 5.0 per cent. These specifications were based on experience and on the early research outlined above.

Today an even greater number of states have specifications governing the percentage of shale in concrete aggregates. A recent survey (5) of the specifications of 46 state highway departments showed 23 with limits on the allowable percentage of shale; 29 with limits on the percentage of "soft fragments" (including shale). Those specifications which imposed no numerical limits generally contained a statement such as: "Coarse aggregate must consist of hard, strong, durable pieces". For those states which had a definite requirement for shale, the percentages allowed varied from 0.5 to 2.0 per cent with most states allowing about 1.0 per cent. The Indiana specifications allow 2.0 per cent shale.

Although most of these specifications have been in effect for quite a few years and are based somewhat on experience and the small amount of research that has been done on shales, there is some doubt as to how realistic these specifications are. For example, none of these specifications take into account differences in types of shales within the area governed by the specification. Also, the percentages of shale allowed are considerably lower than the percentages found not harmful by the Kentucky and Tennessee Highway Departments in their aforementioned studies.

This study of the effects of shale on the freeze-thaw durability of concrete is being carried out to supplement the information obtained in the shale studies of the 1930's, to determine if present-day specifications on shales are realistic, and to suggest any needed changes in the specifications which may become apparent as a result of the laboratory testing.

SHALES INVESTIGATED

The shales tested in this study were all obtained by hand-picking from commercial gravel sources in the State of Indiana. Gravels from five separate deposits were used. Each of these deposits are of glacial origin but they are so widely separated that the shale gravel in each deposit was derived from shale bedrock differing from that serving as a source for the other deposits. Table I lists the shales studied, their geologic origins, and a macroscopic description of the shale from each source. The map in Figure 1 shows the location of the deposits from which the shales were obtained.

IGURE I — LOCATION OF GRAVEL SOURCES FOR SHALE SAMPLES

Table I

LOCATION AND DESCRIPTION OF SHALES

Sample No.	Location	Geologic Source	Description
2063	Decatur	Glacial terrace of St. Mary's River	Hard carbonaceous silty shale
2066	Seymour	Glacial terrace of East Fork of White River	Soft, platey, weak shale
2068	Near LaPorte	Kame on Valparaiso Moraine	Soft argillaceous shale
2075	Indianapolis	Glacial terrace of White River	Fairly hard carbonaceous shale
2076	South Bend	Glacial terrace of St. Joseph River	Weak, platy carbonaceous shale

SCOPE

This report consists primarily of the freezing and thawing test results of concrete beams made from blends of five shale sources with a standard crushed limestone known to have a high durability to freezing and thawing. Blends of 2, 4, 6, and 10 per cent of each type of shale were made with the crushed limestone and these blends were used to make 3 x 4 x 16 - inch concrete beams. These beams were subjected to up to 300 cycles of freezing and thawing in the automatic freeze-thaw machine.

Average ASTM durability factors at 300 cycles of freezing and thawing are reported for the various combinations that were tested. Graphs of values of relative dynamic modulus of elasticity versus cycles of freezing and thawing are also included. In addition, the results of tests to determine the effect of bubble spacing factors and specific surface areas of the air voids on the freeze-thaw durability of air-entrained concrete are presented.

Using this information plus data on physical properties of the shales such as absorption and bulk specific gravity an attempt will be made to reach some qualitative correlation between type and percentage of shale and the amount of disintegration to expect for concrete undergoing freezing and thawing action.

OUTLINE OF WORK

Sampling

Sampling of the shales from the five sources was done by hand-picking from stockpiles. This was extremely slow work since four of the five deposits contained only about 1 to 2 percent shale. The exception was the kame deposit near LaPorte in which approximately 70 percent of the coarse aggregate consisted of shale.

The concrete mixes used in making the test beams were designed to contain equal amounts of four sizes of coarse aggregate: #4-3/8 inch, 3/8 - 1/2 inch, 1/2 - 3/4 inch, and 3/4 - 1 inch. Since the shale was to be blended into the coarse aggregate in definite percentages, it was at first planned to obtain the shale in equal amounts by weight of these four sizes. However, it was soon found to be almost impossible to pick enough of the #4-3/8 inch size, and since previous research (6) has shown the effect of particles 3/8 inch or smaller in size to be negligible in regard to freeze-thaw durability of concrete, it was decided to not use the #4-3/8 inch size of shale. Therefore the hand-picking was restricted to equal amounts of the 3/8 - 1/2 inch, 1/2 - 3/4 inch, and 3/4 - 1 inch sizes.

LABORATORY TESTING

The shale samples were sieved into the three sizes in which they were to be used, and then were tested for bulk specific gravity (saturated, surface-dry) and for 24-hour absorption using the vacuum saturation technique.

A standard mix was designed using crushed limestone coarse aggregate (Source 67-2S), fine aggregate from Source 79-1G, and Type I portland cement (Lab. No. 315). The mix was designed using the Goldbeck version of the $^{b}/b_{o}$ method. The mix design was held constant for all the beams tested except that varying small percentages of shale were substituted for part of the crushed limestone coarse aggregate in all but a few standard beams. In all beams the coarse aggregate was used in equal amounts of the #4-3/8, 3/8-1/2, 1/2- 3/4, and 3/4-1 inch sizes. For the fine aggregate, two parts of the 0 - #16 size were used for each part of the #16 - #4 size.

A water - cement ratio of 0.46 by weight was used. This water-cement ratio produced a mix with good workability and a slump of about 3 inches. The cement factor was kept constant at 6 bags per cubic yard. Darex was used to entrain approximately 4 percent of air in each batch. Air contents were determined gravimetrically, using a 0.1 cubic foot measure for the unit weight test. The concrete was molded into 3 x 4 x 16-inch beams and these beams were cured by immersion in lime water for 13 days following removal of the specimens from the molds one day after casting.

An experimental outline was set up in which three variables were introduced into the production of these beams with all other factors to remain as constant as possible. The three variables were:

(a) The five different sources of shale. Material from each of these sources was blended with the crushed stone coarse aggregate in different beams.

(b) The percentage of shale to be included in the coarse aggregate. Shale from each of the sources was added to the crushed stone in blends of 2, 4, 6, and 10 percent.

(c) Type of mixing. Beams made from all possible combinations of coarse aggregate were mixed both by hand and by means of a small power mixer. This variation was introduced because it was noted early in the testing program that those beams mixed by hand failed much more rapidly when exposed to freezing and thawing than did those mixed by machine. Henceforth several sets of machine-mixed beams were duplicated by hand-mixed beams to try to determine by laboratory techniques the reason for this difference in durability.

All other factors such as type of cement, water-cement ratio, mixing time and mixing procedure, etc., were kept as constant as possible to avoid the introduction of any additional variables. For each combination of variables 3 or 6 beams were made. To insure an equal amount of shale in each beam, the concrete for each beam was batched and mixed separately. The 3/8 - 1/2 inch and the 1/2 - 3/4 inch pieces of shale were blended with the crushed stone coarse aggregate before mixing and thus were randomly spaced in the resulting beam. Since the 3/4 - 1 inch pieces of shale were the largest and thus thought to be the most important in destruction of the concrete due to freezing and thawing, and since only a few of these pieces were used in each beam, it was decided that the position of these few pieces in each beam should be controlled.

Thus the 3/4 - 1 inch pieces were placed in a row at mid-depth along the longitudinal axis of the beam. If one piece was used, it was placed right at the center of the beam; if two were used, they were placed at the one-third points; etc.

The freezing and thawing exposure was carried out in the automatic freeze-thaw machine. Approximately six cycles per day of the fast freeze in air, thaw in water cycle were obtained (ASTM C291-57T). Periodic determinations of the relative dynamic modulus of elasticity of the beams were made to measure the amount of deterioration of the concrete. The end point for the exposure adopted in this test series was 50 percent relative dynamic modulus of elasticity or 300 cycles of freezing and thawing, whichever occurred first. Using the data obtained from the freeze-thaw testing, curves were plotted for each group of beams relating the number of cycles of freezing and thawing undergone to the dynamic modulus of elasticity for each beam as determined by sonic means. Also, a durability factor as defined in ASTM C291-57T, and based on 300 cycles of freezing and thawing, was computed for each beam.

During the freeze-thaw testing program considerable pitting of the surfaces of the beams occurred. Therefore visual observation of all pitting was conducted on each beam every 10 - 20 cycles. The size of each pit was noted along with the size and type of the piece of aggregate causing the failure.

In an effort to determine the reason for the notable difference in durability between the hand-mixed and machine-mixed beams, a subsequent study was made of the character of the air voids in several of the beams by means of the linear traverse technique outlined by Fears (7). It was hoped that a comparison of the air bubble size and spacing for the hand-mixed and machine-mixed beams might provide laboratory proof of the

theoretical concept developed by Powers (8). This concept stipulates that the durability of air-entrained concrete is inversely related to the air-void spacing factor of the concrete and directly related to the specific surface area of the air voids in the concrete.

TEST RESULTS

A summary of the results of the bulk specific gravity tests on the shales is shown in Table II. These data are presented graphically in Figure 2.

Table III presents the results of the vacuum absorption tests.

Table II

Bulk Specific Gravity Values for Shale Samples

Bulk Specific Gravity	Percent of Shale Sample in Each Specific Gravity Range				
	2063 (Decatur)	2066 (Seymour)	2068 (LaPorte)	2075 (Indianapolis)	2076 (South Bend)
2.55 plus	3.9%	0.0%	0.0%	1.6%	0.0%
2.45-2.55	6.5	0.0	0.0	2.9	0.0
2.35-2.45	23.8	0.9	0.3	26.1	0.0
2.25-2.35	40.5	7.3	1.3	41.6	12.2
2.15-2.25	13.4	25.2	56.4	19.4	67.3
2.05-2.15	8.9	36.4	39.7	6.1	20.4
2.05 minus	2.9	30.1	2.4	2.2	0.1

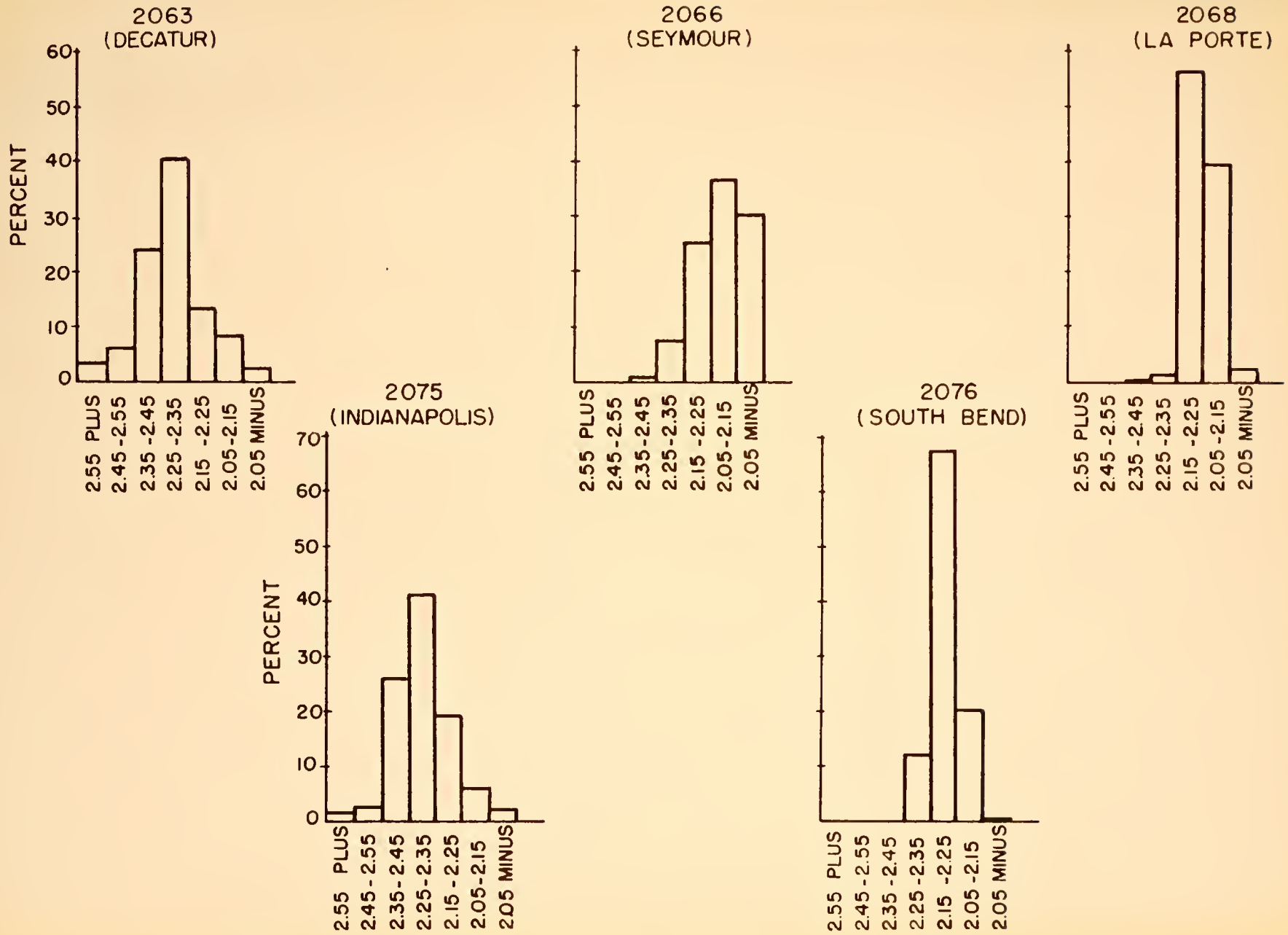

FIGURE 2-BULK SPECIFIC GRAVITIES FOR FIVE SHALE SAMPLES

Table III

Percent Absorption of Shale Samples as Determined by

24-Hour Vacuum Saturation

Shale Sample				
2063 (Decatur)	2066 (Seymour)	2068 (LaPorte)	2075 (Indianapolis)	2076 (South Bend)
1.7%	7.1%	12.6%	4.1%	8.1%

A summary of the test results for the freeze-thaw testing is shown in Table IV. This table shows the data on mixes separated into cells each of which is comprised of a different combination of shale source, percentage of shale, and type of mixing. Within each cell are shown durability factors* based on 300 cycles of freezing and thawing. Also shown within each cell is the mean value of the durability factors for that cell. Since individual beams sometimes result in rather erratic results, these average durability factors are probably more indicative of the durability of the concrete represented by a particular cell than are the individual durability factors. It should be noted, however, that in the light of past experience with this sort of testing, the variability in the individual results is not excessive.

The freezing and thawing test results are presented graphically in Figures 3 - 7. There is a graph for each shale source and each curve on every graph represents an average of the freeze-thaw curves within a particular cell in which a certain percentage of shale was used.

In the visual study of surface failure of the beams considerable pitting was noted on the surfaces of some of the beams. Approximately 90 percent of the pits appeared to have been caused by shale pebbles. There was some relationship between the percentage of shale in each beam and the amount of pitting occurring on the surface of the beam. Also, it was noted that for the same percentages of shale, hand-mixed beams had more surface deterioration than machine-mixed beams. Apparently these surface failures had little effect on the strength of the beams because beams which had experienced no loss of dynamic modulus sometimes were pitted.

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*Durability Factor (DF) $= \frac{PN}{M}$ where:

P = relative dynamic modulus of elasticity at N cycles, percent
N = number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and
M = specified number of cycles (300) at which the exposure is to be terminated.

Table IV

Summary of Individual Average ASTM Durability Factors for Freeze-Thaw Testing Program of Concrete Beams Containing Small Percentages of Shale Coarse Aggregate

Percent of Shale in Coarse Aggregate	Shale Sample									
	2063 (Decatur)		2066 (Seymour)		2068 (LaPorte)		2075 (Indianapolis)		2076 (South Bend)	
	Machine Mixed	Hand Mixed	Machine Mixed	Hand Mixed	Machine Mixed	Hand Mixed	Machine Mixed	Hand Mixed	Machine Mixed	Hand Mixed
2%	100.0	32.0	97.8		99.5	72.5	96.5	63.0	96.0	
	98.8	28.0	96.0		85.5	42.6	48.9	15.8	96.5	
	92.0	55.0	92.8		94.0	38.2	88.5	14.0	96.0	
		41.6	94.5			24.7			95.4	
		48.6	95.3			36.6			95.8	
		86.0	62.3			13.7			96.3	
	Ave.=96.9	Ave.=48.5	Ave.=89.8		Ave.=93.0	Ave.=38.1	Ave.=78.0	Ave.=30.9	Ave.=96.0	
4%	96.3	37.0	96.5		94.3	40.6	96.4	71.4	96.3	
	85.3	50.5	78.5		43.0	95.0	96.4	27.6	94.0	
	97.1	21.0	90.8		44.2	22.6	97.2	39.0	97.2	
			80.5			23.6			93.0	
			26.3			27.6			96.0	
			87.2			19.1			99.5	
	Ave.=96.2	Ave.=36.2	Ave.=76.6		Ave.=60.5	Ave.=38.1	Ave.=96.7	Ave.=46.0	Ave.=96.0	
6%	99.0		97.4		42.4		98.5	21.4	99.8	
	98.2		92.5		98.4		72.4	15.9	89.0	
	96.0		98.2		44.6		96.5	10.6	100.0	
	42.4		96.3		87.9				97.0	
	98.7		87.6		72.5				89.0	
	100.0		96.5		41.7				88.4	
	Ave.=89.1		Ave.=94.7		Ave.=64.5		Ave.=89.1	Ave.=16.0	Ave.=93.9	
10%	95.5	6.0	87.0	29.4	27.2	8.3	52.0	5.3	90.8	28.8
	86.5	15.3	96.5	14.2	83.0	15.3	98.0	9.7	95.4	30.1
	95.0	25.6	33.8	29.3	47.6	14.8	96.2	9.5	95.5	17.5
	Ave.=92.3	Ave.=15.6	Ave.=72.4	Ave.=24.3	Ave.=52.6	Ave.=12.8	Ave.=82.1	Ave.=8.2	Ave.=93.9	Ave.=25.5

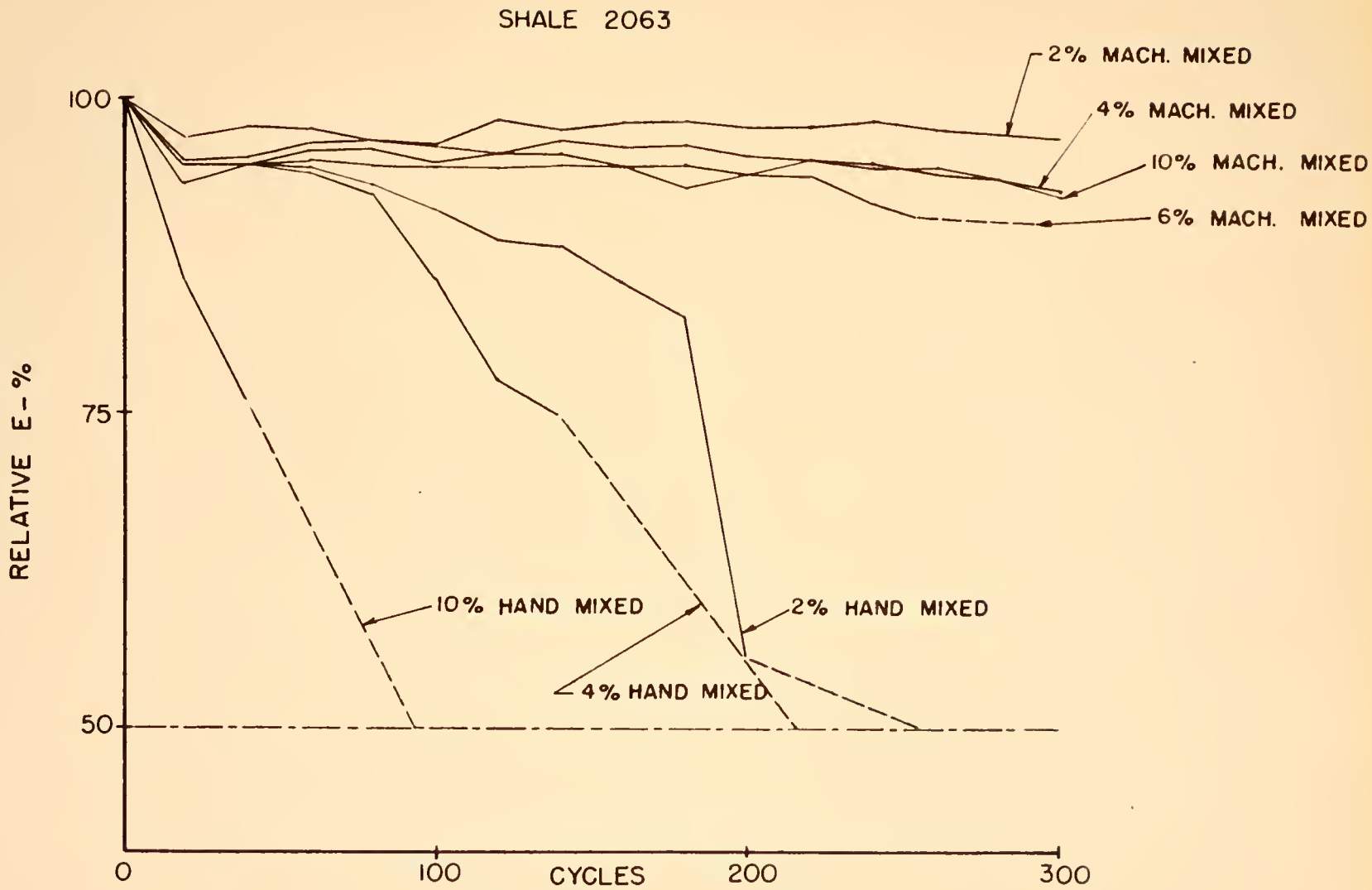

FIGURE 3- AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS MADE FROM NO. 2063 SHALE

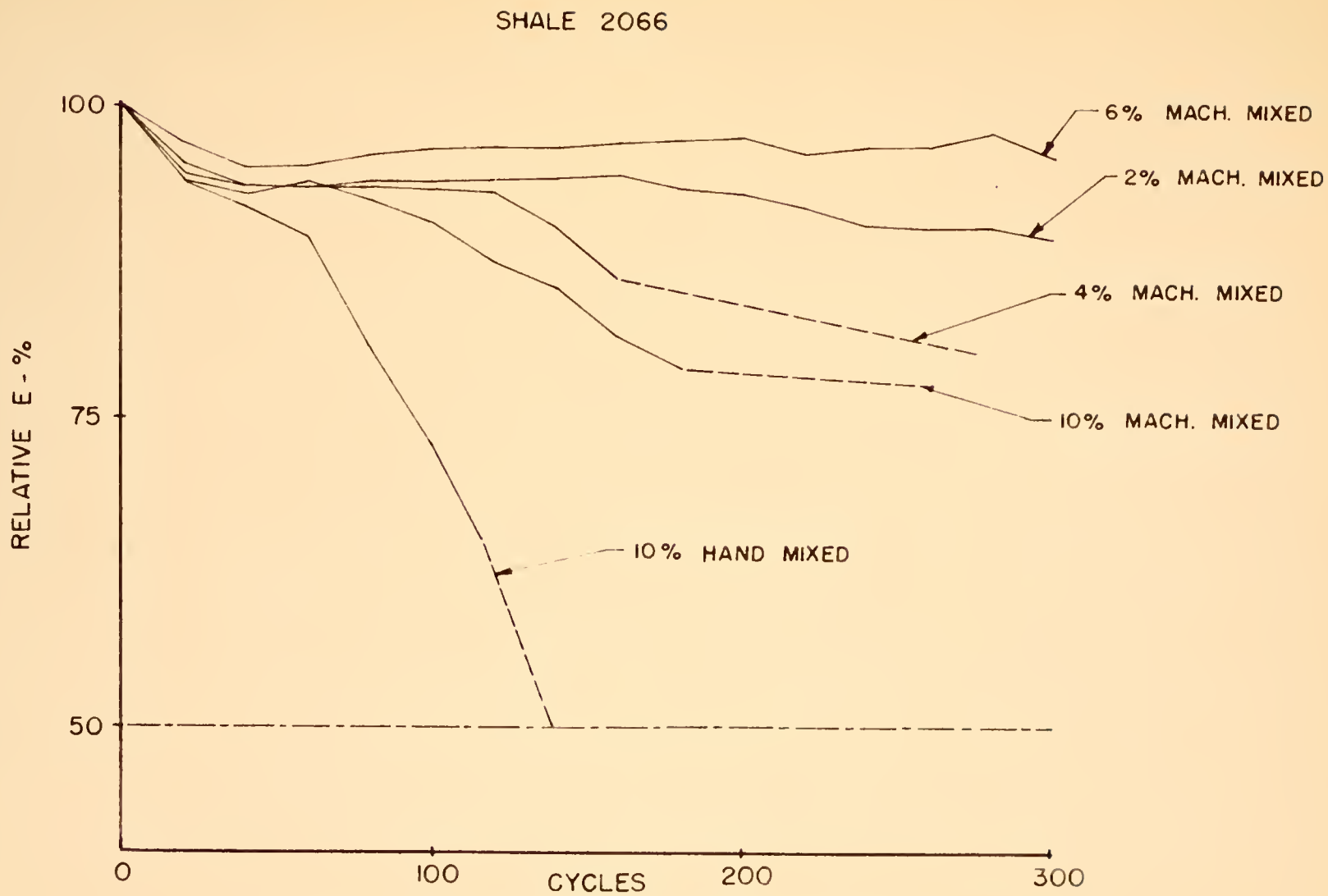

FIGURE 4 - AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS MADE FROM NO. 2066 SHALE

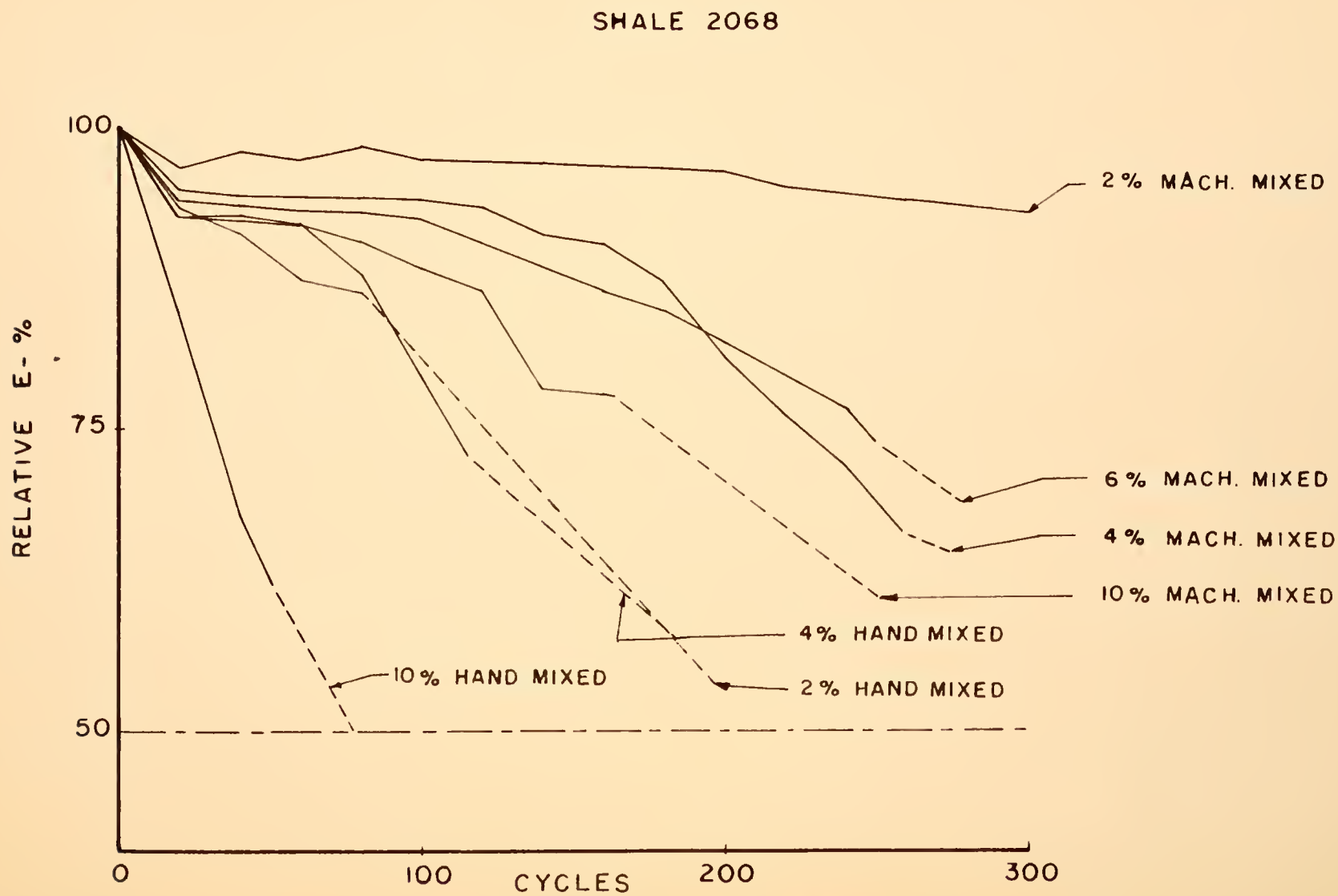

FIGURE 5-AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS MADE FROM NO. 2068 SHALE

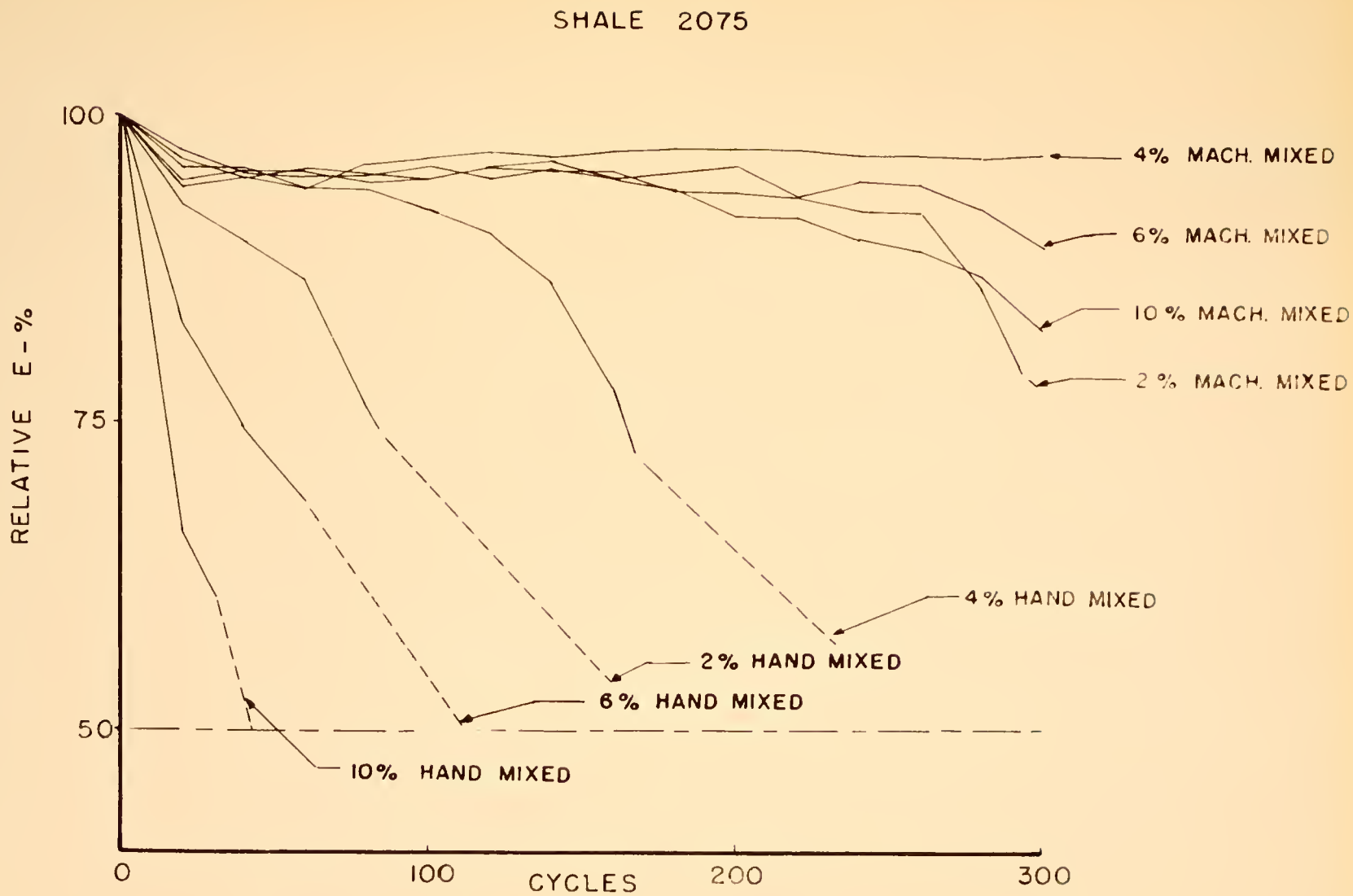

FIGURE 6-AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS MADE FROM NO. 2075 SHALE

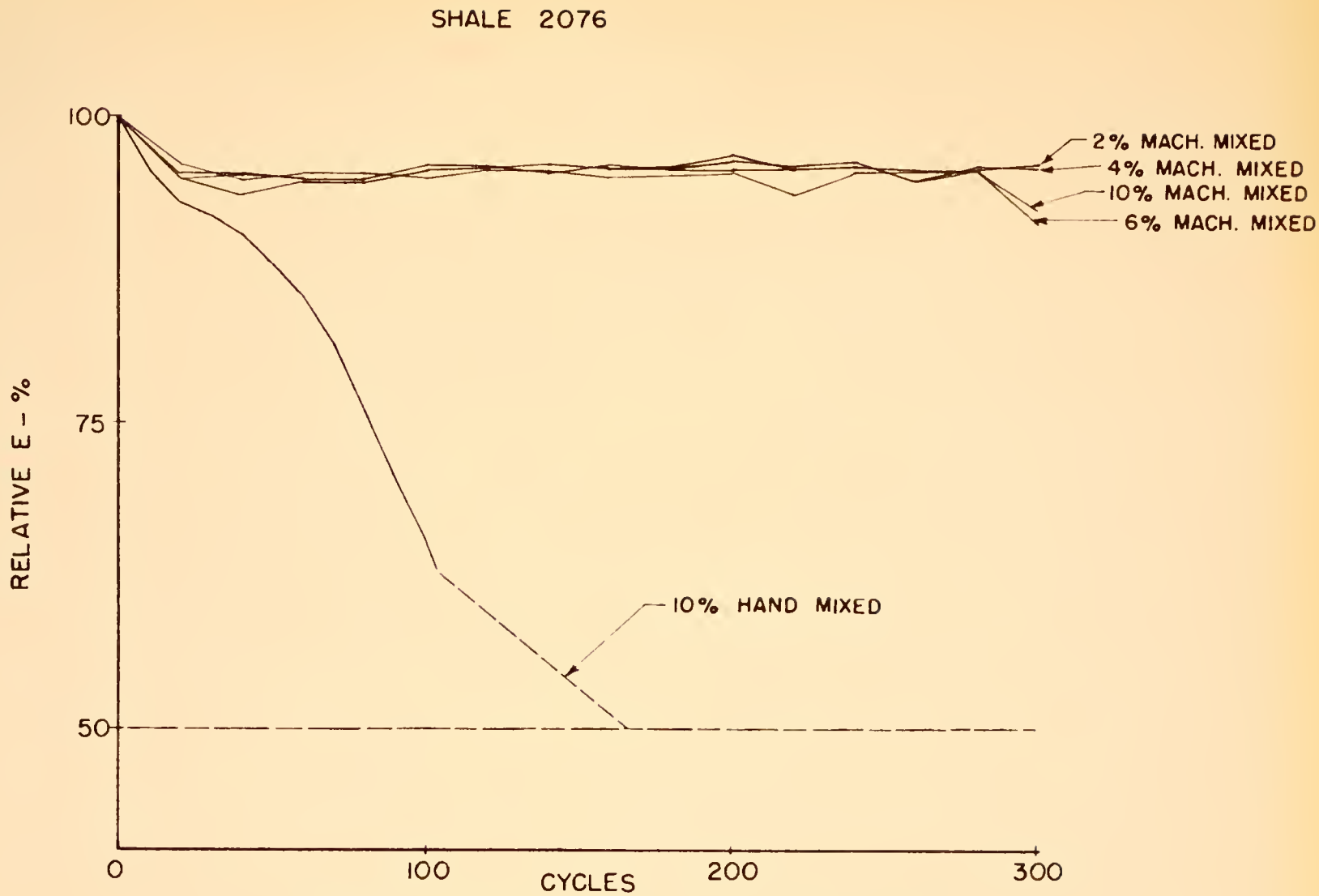

FIGURE 7 - AVERAGE FREEZE-THAW CURVES FOR CONCRETE BEAMS MADE FROM NO. 2076 SHALE

Linear Traverse Tests

Table V presents the results of the study of air voids in certain beams by means of the linear traverse technique. Figure 8 presents the same data graphically. For this study pairs of beams were chosen in which both beams had identical compositions, but one was hand-mixed and the other was machine-mixed. In all cases the hand-mixed beams produced lower durability factors than did the machine-mixed beams. By means of the linear traverse technique, the total percentage of entrained and entrapped air as well as the bubble spacing was determined for each beam. As recommended by Fears, a 200-inch traverse was run on each beam. Using the values obtained for total percent of air and voids per inch of traverse, it was possible to compute specific surface areas and bubble spacing factors for the beams. The specific surface areas were computed as follows:

α = specific surface area of voids = $\frac{4n}{A}$ where

n = voids per inch

A = total air content in percent

The bubble spacing factors were computed as follows:

L = spacing factor = $r_m - r_h$ where

r_m = radius of "sphere of influence" = $\frac{\sqrt{3}}{2}\left(\frac{p + A}{N}\right)^{1/3}$

p = paste content per unit volume of mix

A = total air content in percent

N = hypothetical voids per cubic inch = $\frac{A\alpha^3}{36\pi}$

r_h = radius of hypothetical sphere = $\frac{3}{\alpha}$.

An example of the use of these equations can be presented by going through the calculations needed to arrive at the values in the

Table V

Results of Air Void Studies of Certain Concrete Beams by Means of the Linear Traverse Technique

Beam Number and Description	Durability Factor	% Air	Voids per Inch	Calculated Specific Surface Area of Voids (sq.in./cu.in.)	Calculated Void Spacing Factor (Inches)
S6-3 (4% Shale #2063, machine mixed)	97.1	3.3	4.2	506	.008
S6-6 (4% Shale #2063, hand mixed)	21.1	4.1	2.9	287	.013
S8-6 (10% Shale #2063, machine mixed)	95.0	3.8	4.8	512	.007
S8-1 (10% Shale #2063, hand mixed)	6.0	3.8	2.0	208	.018
S9-5 (10% Shale "2068, machine mixed)	83.0	2.7	3.5	523	.009
S9-1 (10% Shale "2068, hand mixed)	8.3	3.3	2.7	324	.013
S20-2 (10% Shale "2066, machine mixed)	96.5	3.5	3.8	441	.009
S20-5 (10% Shale #2066, hand mixed)	14.2	3.0	2.0	269	.017
S23-3 (10% Shale #2075, machine mixed)	96.2	3.1	5.0	637	.007
S23-4 (10% Shale #2075, hand mixed)	5.3	3.8	3.0	316	.012

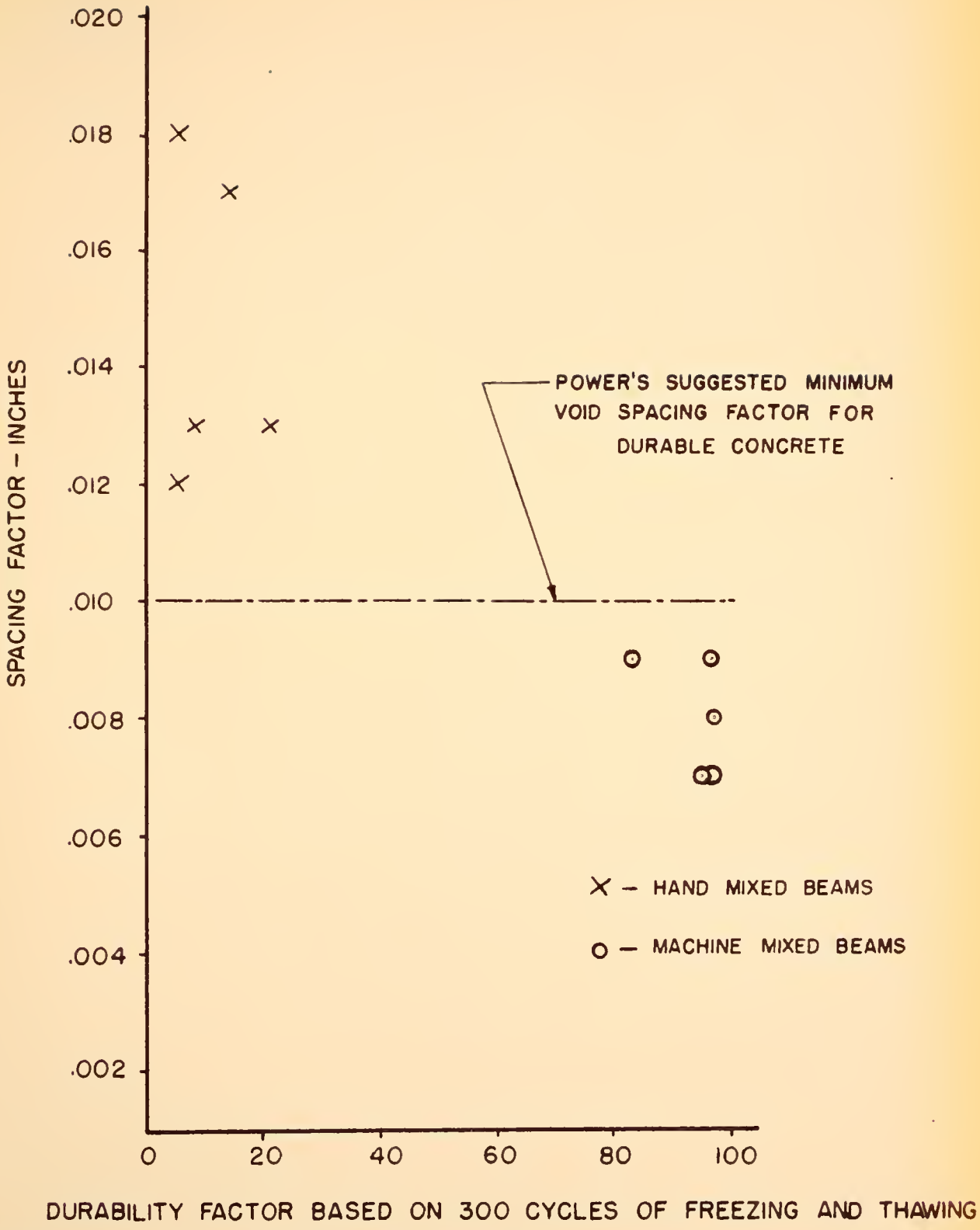

FIGURE 8 - RELATIONSHIP OF VOID SPACING FACTOR TO DURABILITY OF AIR-ENTRAINED CONCRETE

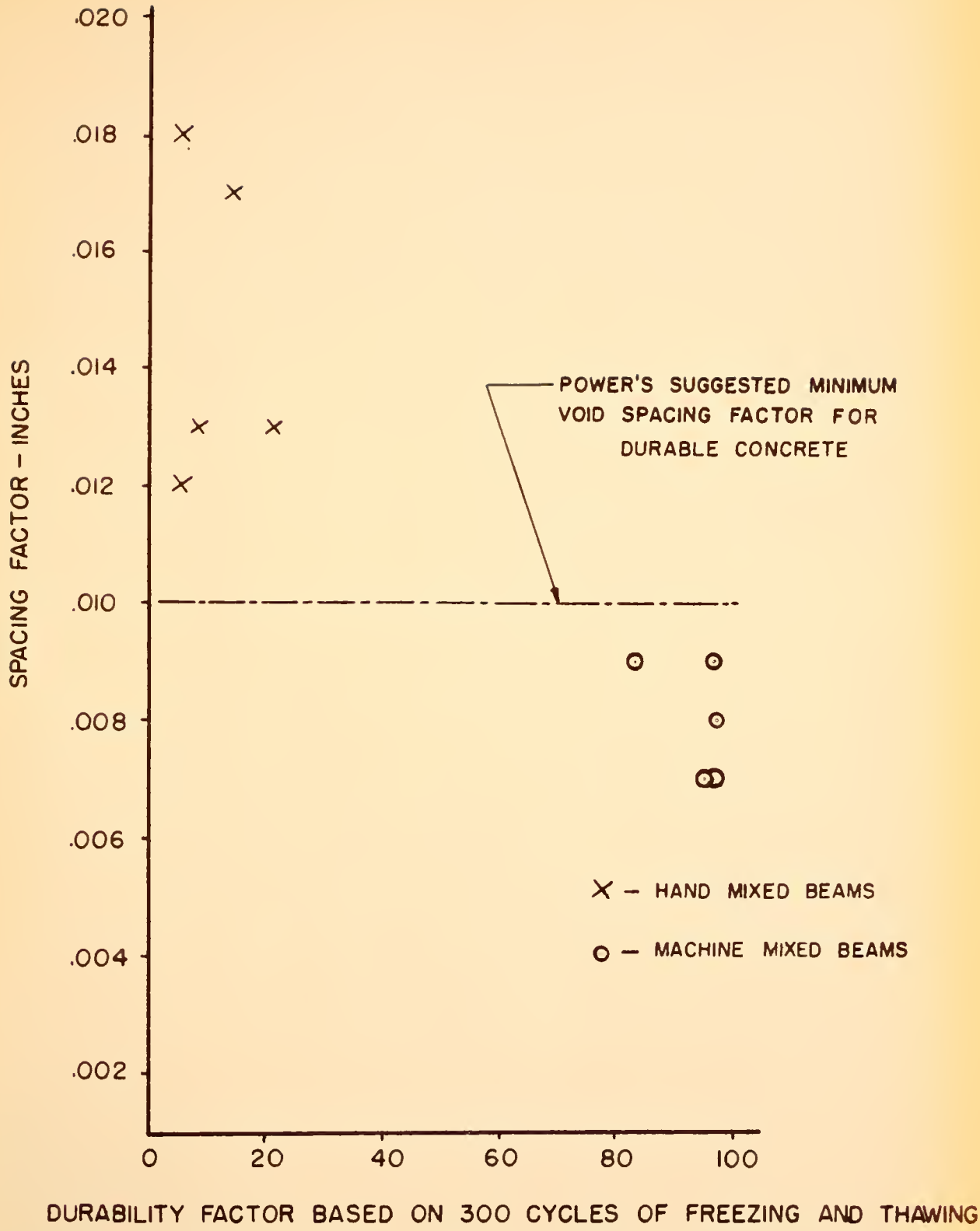

FIGURE 8 - RELATIONSHIP OF VOID SPACING FACTOR TO DURABILITY OF AIR-ENTRAINED CONCRETE

first line of Table V. By means of the linear traverse technique it was found that the concrete in beam S6-3 contained 3.32 percent air voids and had an average of 4.20 voids per inch of traverse. The calculations were as follows:

Specific surface area of voids, $\alpha = \frac{4n}{A} = \frac{(4)(4.20)}{(.0332)} = 506$ sq.in./cu.in.

Hypothetical voids per cu.in., $N = \frac{A\alpha^3}{36\pi} = \frac{(.0332)(506)^3}{36\pi} = 38{,}000$

Paste content per unit volume of mix, $p = .126$ (from mix design)

Radius of hypothetical sphere, $r_h = \frac{3}{\alpha} = \frac{3}{506} = .00593$

Radius of "sphere of influence", $m = \frac{\sqrt{3}}{2}\left(\frac{p + A}{N}\right)^{1/3} = \frac{\sqrt{3}}{2}\left(\frac{.126 + .0332}{38{,}000}\right)^{1/3} = .01397$

Spacing factor, $L = r_m - r_h = (.01397 - .00593) = 0.008$

DISCUSSION OF RESULTS

The histograms in Figure 2 show that shales #2063 and #2076 have higher bulk specific gravities than the other three shale samples. Also, shale #2066 has the lowest bulk density of any of the samples with 30 percent of this shale falling below a bulk specific gravity of 2.05.

In general, there is a fairly well-pronounced relationship between the bulk specific gravity of these shales and their absorptions. As would be expected the denser samples generally had the lowest percentages of absorption. Shales #2063 and #2075 with the highest bulk densities had the lowest absorptions, 1.7 and 4.1 percent respectively. Shales #2066, #2068, and #2076 which have fairly low bulk specific gravities have fairly high absorptions of 7.1, 12.6, and 8.1 percent, respectively. Note that although shale #2068 is not the lightest of the samples, it does have the highest absorption value.

Several interesting points may be brought out from the freeze-thaw data summarized in Table IV and Figures 3 - 7. First, for the machine-mixed beams made with shales #2063, #2066, #2075, and #2076, there seems to have been very little loss of freeze-thaw durability caused by increasing the percent of shale from 2 percent to 10 percent. For shale #2068 which has an absorption of 12.6 percent, this does not seem to hold true. An apparent increase in deterioration of the concrete in which shale #2068 was used occurred as the percentage of shale was increased from 2 percent to 10 percent. It is difficult to tell exactly at what percentage deterioration might become "critical" for this shale, but there is a definite drop in the average durability factor in going from 2 percent to 4 percent. It should be noted here that besides being the most absorptive of the five shales tested, #2068 was also the softest and the most argillaceous. It split into layers when soaked in water and the individual fragments had very little strength when in a saturated condition. Unlike the other shales tested, shale #2068 is from a gravel which is seldom, if ever, used for concrete aggregate. This gravel is from a farm pit a few miles west of LaPorte and is used by the county for road metal.

The data show a definite difference in durability between the machine-mixed and the hand-mixed beams. For every case in which beams with the same percentage of the same shale were mixed by both methods, the machine-mixed beams proved to be considerably more durable than the hand-mixed. This was thought to have been caused by the better distribution of entrained air to be expected with machine-mixing. Also, unlike most of the machine-mixed beams, the hand-mixed beams showed a definite drop in durability in going from 2 percent shale to 10% shale for the three shale types from which a full series of hand-mixed beams were made.

This seems to indicate that if the cement paste is not well-protected by proper air-entrainment, percentages of shale from 2 to 10 percent can cause a definite reduction of the durability of the concrete to deepseated freeze-thaw damage. Conversely it seems that concrete which is protected by proper air-entrainment can contain at least up to 10 percent of most shales without undergoing much deepseated damage when exposed to freezing and thawing except for some surface pitting.

It should be noted that these data are not statistically homogeneous. In several of the cells of Table IV, there are individual durability factors which differ considerably from the other factors in the same cell. As stated earlier, in the light of past experience with this sort of testing, the variability in these results is not excessive. However, in those cells which include results from only three beams, extreme variability within the results probably will necessitate making three more beams in order to obtain a more complete statistical picture. After producing and testing the extra beams required to provide the additional data, a complete analysis of variance will be performed so that more quantitative conclusions can be reached.

The results of the study of air content of the hardened concrete apparently confirm the theoretical air void concepts developed by Powers (8). Powers theorized that the increase in durability afforded concrete by means of air entrainment is largely a function of the spacing of the air voids in the concrete. He suggested that a concrete containing air voids with high specific surface area, and thus with a low void spacing factor, would receive more protection from the air voids than one with low specific surface area and high spacing factor. He considered a void spacing factor of about 0.01 inches to be critical; those concretes

with spacing factors lower than 0.01 inches were thought to be well protected from freezing and thawing deterioration; those with spacing factors greater than 0.01 inches were thought to be poorly protected.

This theory is supported by the results of the linear traverse studies as summarized in Table V and Figure 8. For the few beams studied it was found that those with high durability factors all had spacing factors below 0.01 and those with low durability factors had spacing factors above 0.01. Also, the specific surface areas of the beams with high durability factors were all higher than those with low durability factors. Of course these studies were run on only a few beams and several more beams will have to be studied before definite conclusions are reached.

As previously mentioned considerable pitting took place on the surfaces of some of the beams during the freeze-thaw testing program. There was some relationship between the percentage of shale in the beams and the amount of surface deterioration which took place.

The study of surface deterioration also showed clearly that the machine-mixed beams had undergone less pitting than the hand-mixed ones. In fact many of the beams which contained 6 or 10 percent shale but were machine-mixed showed almost no surface deterioration while their hand-mixed equals were badly pitted. This difference in the amount of surface deterioration is probably due to the better distribution of entrained air in the machine-mixed beams.

It should be noted that surface deterioration apparently had little effect on the dynamic modulus of elasticity of the beams. Some beams deteriorated rather badly on the surface with very little loss of strength. Surface deterioration, therefore, probably does little structural damage to the concrete.

SUMMARY OF RESULTS

The following is a summary of the important findings thus far:

(1) For properly air-entrained concrete four of the five shales tested caused practically no loss in freeze-thaw durability when used in amounts of up to 10 percent in concrete exposed to 300 cycles of freezing and thawing.

(2) For properly air-entrained concrete the only notable loss of durability due to 300 cycles of freezing and thawing occurred in a concrete made with 4 to 10 percent of a shale with an absorption of 12%. This would indicate that highly absorptive shales may cause trouble when used in concrete exposed to freezing and thawing.

(3) Hand-mixed concrete is not as durable to deterioration caused by freezing and thawing as machine-mixed concrete. This is probably due to the better distribution of entrained air achieved by machine mixing. The results of studies of air voids in both machine-mixed (durable) and hand-mixed (non-durable) beams seem to support Powers' theory that a concrete containing voids with a high specific surface area and a low void spacing factor is more resistant to freeze-thaw deterioration than one whose voids have a low specific surface area and a high spacing factor. Also, these air void studies appear to support his theory that a void spacing factor of about 0.01 inches is critical; i.e., those concretes with spacing factors less than 0.01 inches appear to be well protected from deterioration due to freezing and thawing

while those with spacing factors greater than 0.01 inches are poorly protected.

(4) For all five of the shale types, percentages of shale of 2 - 10 percent in concrete tends to lower the durability of the concrete to freezing and thawing if the concrete is not properly air-entrained. Also, as the percentage of shale increases from 2 - 10 percent for beams which have poor air entrainment, the durability of the concrete decreases.

(5) All five shale types caused some surface pitting of the concrete beams. The surface deterioration was most evident in the hand-mixed beams and increased somewhat with increase in the percentage of shale.

FURTHER WORK TO BE DONE IN SHALE STUDY

The following steps are planned to complete the shale study:

(1) More machine-mixed beams will be made for some aggregate combinations in order to complete the picture on freeze-thaw durability. The new beams will be made mainly for those cells of the experimental data layout which contain only three readings, one of which varies considerably from the other two.

(2) A complete analysis of variance will be made of the final freeze-thaw data.

(3) Mineralogical analyses of the five shale samples will be carried out. Microscopic analyses have already been attempted, but haven't been too successful because of the

very small grain size of the shales. Therefore, subsequent analyses will attempt the use of differential thermal analysis and x-ray diffraction.

(4) The study of air-void characteristics by the linear traverse technique will be continued.

(5) A statistical study will be carried out to attempt to determine a quantitative relationship between the amount of surface deterioration caused by shale and the percentage of shale in the concrete.

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